

Lawrence Berkeley National Laboratory
Advanced Light Source – Beamline 1.4



INTERNSHIP REPORT

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About LBNL

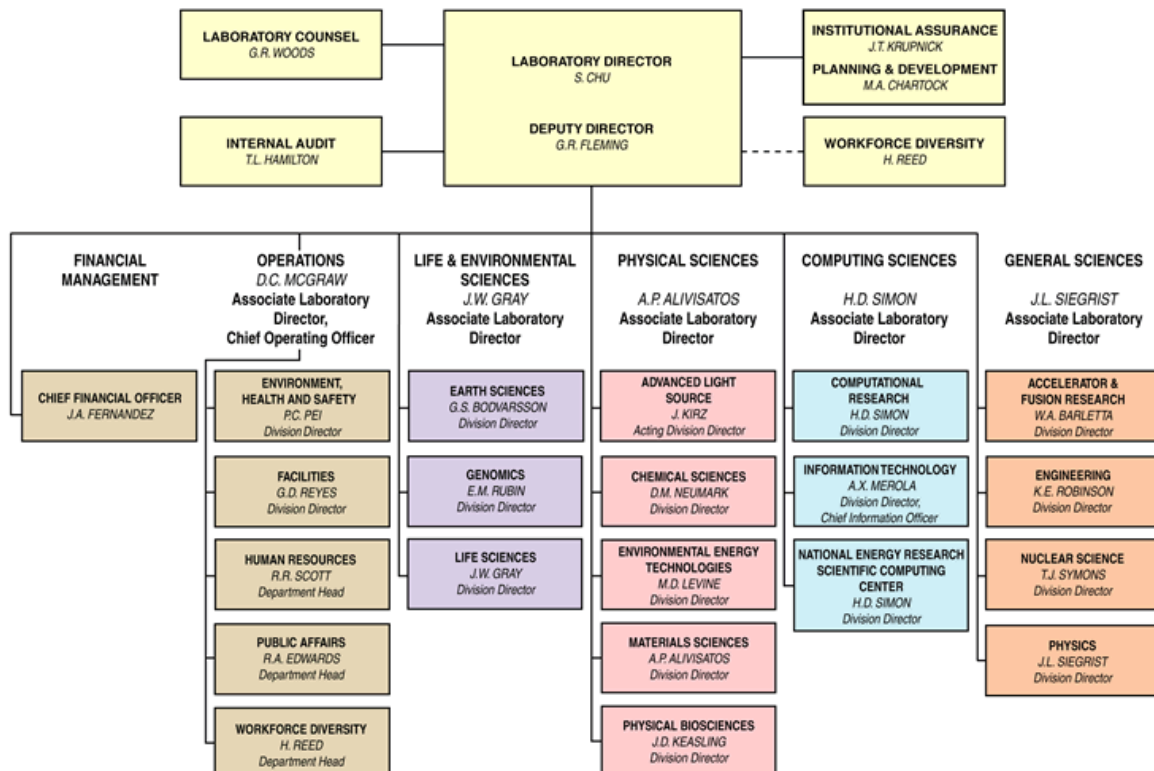
The LBNL

The Lawrence Berkeley National Laboratory (LBNL) was the first national laboratory. It is operated for the Department of Energy (DOE) and is managed by the University of California. LBNL was founded in 1931 by Ernest Orlando Lawrence, winner of the 1939 Nobel Prize in physics for his invention of the cyclotron, a circular particle accelerator that opened the door to high-energy physics.

LBNL is organised into 17 scientific divisions and hosts four DOE national user facilities including the Advanced Light Source, the National Energy Research Supercomputing Center, the National Center for Electron Microscopy, and the 88-inch Cyclotron.

Berkeley Lab's mission includes the following: to perform multidisciplinary research in the energy sciences, general sciences and bio-sciences; to develop and operate unique national experimental facilities; to educate and train future scientists; to transfer knowledge and technical innovations between LBNL research programs, universities and industry.

LBNL is currently directed by Steven Chu.



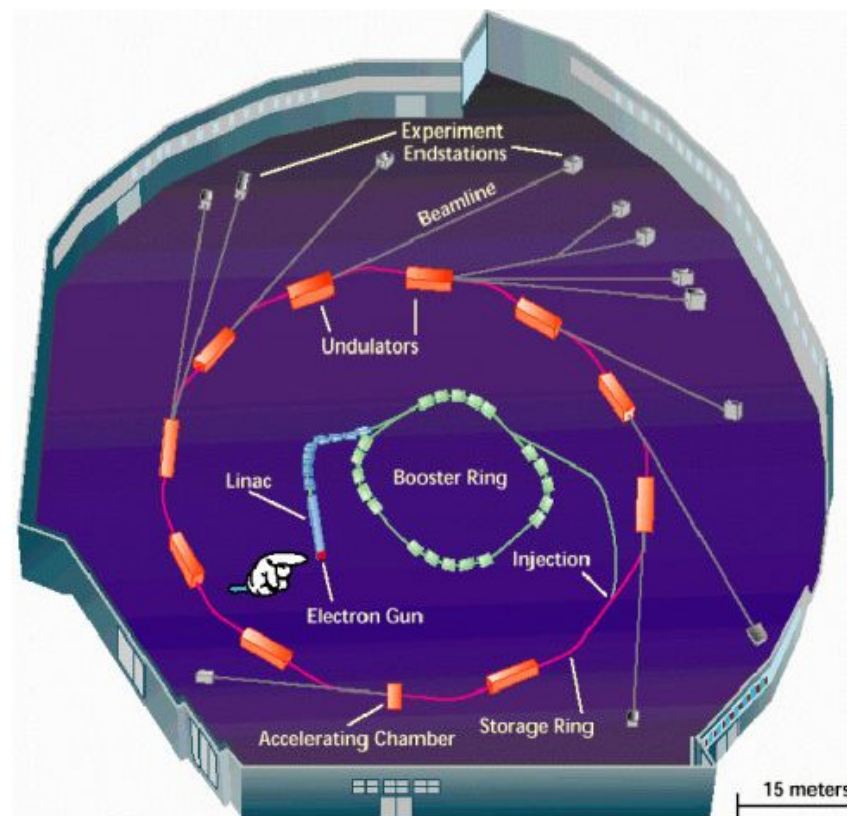
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The ALS

The Advanced Light Source (ALS) is one of the 4 national research facilities at LBNL. It produces intense light whose energy ranges from infrared to x-rays thanks to a synchrotron which will be described later. Thus, scientists can study the properties of materials, probe the structure of atoms or molecules, analyze samples for trace elements, and so on ...

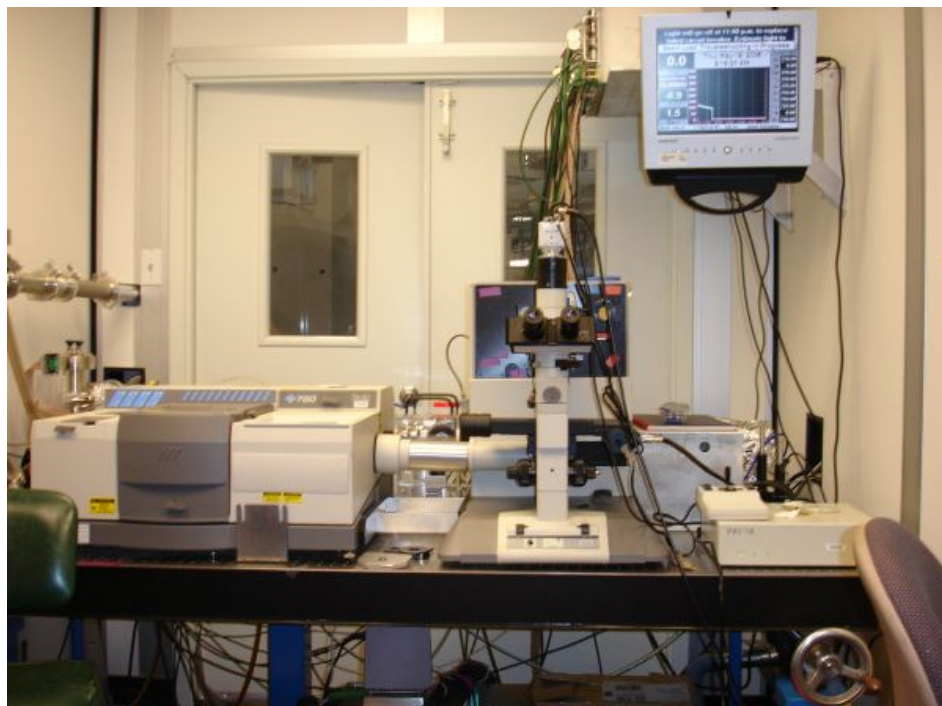
The main part of the synchrotron is the electron storage ring. It's a vacuum chamber which holds an electron beam traveling through it at nearly the speed of light and at high energy. The storage ring is roughly circular with 12 arc-shaped sections joined by 12 straight sections. As the electron beam curves in a magnetic bending section of the ring, it emits light collected by beamlines which deliver the light to the experiment stations. Many straight sections have undulators which are sources of narrow bandwidth high-brightness soft x-rays.

One advantage of this bright source is that many scientists can use the synchrotron at the same time. The lifetime of the beam in the ring is long; after 8 hours approximately $\frac{1}{2}$ of the beam current is lost so the ring is refilled. The electron beam is physically small which means that the light emitted can be refocused to a very small area. So, scientists can study smaller objects.



Beamline 1.4

As said above, scientists use light of different frequencies to study materials. Most of them use x –rays but beamline 1.4 uses light in the infrared region for experiments in environmental, biological, and material sciences.



Scientists study samples using transmission or reflection properties to obtain a spectrum of their sample. The synchrotron IR beam is focused to a small spot (< 10 microns) which allows the study of small samples.

The units mainly used for infrared spectroscopy are wavenumbers instead of wavelength. Wavenumber is the inverse of wavelength in centimeters. So wavenumber is proportional to the energy of the photons.

Introduction

The beamline1.4 has acquired a cryostat designed by the firm Janis but hasn't had time to test it so far. It is supposed to reach a very low temperature, around 0.3 K. Some samples need to be cooled to such temperatures so that they are worth studying. That's the case for semi-conductors with a very small band gap. If we want to study it when it behaves as an insulator, we have to cool it down; otherwise, room temperature generates enough energy so that electrons can jump through the band gap and the material shows some conductivity. Some other materials such as superconductors should be studied at low temperature too since superconducting transition temperatures can be very low.

The cryostat has two optical accesses and different windows can be used. Each window will transmit different wavelengths of the light. Scientists can carry out transmission or reflection measurements. Obviously, the sample will be affected by room temperature radiation, especially when a transmission measurement is in place since there will be windows on two sides of the sample.

I didn't know much about infrared spectroscopy. I've been studying spectroscopy in the gamma or X-ray region. During my stay at the ALS, I was showed some basics of infrared spectroscopy, but I mainly work on this cryostat. My work was to test the cryostat and see what is the lowest temperature we could reach and what results we should expect for different windows. Afterwards, I should be able to write down a manual with things to do and to avoid for future users of this cryostat.

Last year, Jason Singley, a former post-doc, and Zhao Hao, a current post-doc, flew to Boston to see the cryostat functioning at the Janis factory. Michael Martin had also designed a table on which the cryostat would be mounted. I first designed some pieces so that the cryostat will be safe and stable on this table and read some information about cryostats and cryogenic techniques. Then I performed some tests on the cryostat. We cooled it down twice, in different configurations. The first time, we closed the optical accesses. Then we opened both so that the cryostat was in the transmission configuration. We used polyethylene windows which transmit light below 700 cm^{-1} . During this second cool down, we met some troubles but we managed to obtain some results. 2 configurations were tested during this second cool down: aluminium was folded around the windows and then removed. Finally, we tried to calculate the heat load through the windows and compared the results to the measured holding times we obtained. Afterwards, I tried to generalize the calculations so that we can estimate what performance we should expect for others types of windows.

I used Origin 6.0 to obtain the graphs and Dev-C++ to code some C programs for my calculations.

1. The He-3 cryostat: how does it work?

1.1. some physics principles

Consider a liquid in a beaker. It is always evaporating to generate vapor which will be in equilibrium with this liquid. And the vapor is always condensing to be in equilibrium with the liquid. At equilibrium, the rate of evaporation and condensation are equals. So if we remove some vapor, the liquid will evaporate more to re-established the equilibrium.

When it is evaporating, the liquid needs energy. This energy will be taken from the beaker. As a result, the beaker will be cooled down.

Heat can be transferred by 3 different ways: conduction, convection, and radiation.

To keep a cryostat cool, we want to avoid those heat transfers. If we are in vacuum, conduction and convection can't take place. To eliminate radiation from room temperature, we can use good reflection materials or shield the cryostat from room temperature with various heat shields which are at colder temperatures.

Nitrogen gas condenses at 77 Kelvin and freezes at 63.

Liquid He-4 condenses at 4.2 Kelvin. Helium does not freeze at atmospheric pressure.

Helium-3 condenses at 3.2 Kelvin.

1.2. the Janis He-3 cryostat

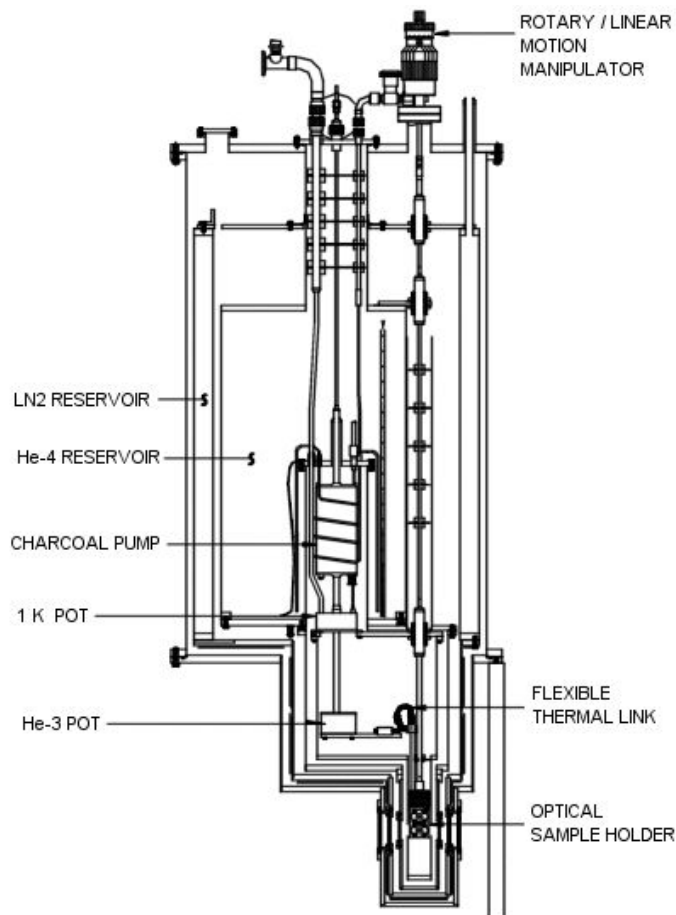
The cryostat consists of three reservoirs inserted one in each other.

The outer reservoir sealed the cryostat and is under vacuum. This reservoir is at room temperature and consequently emits radiation at 300 K.

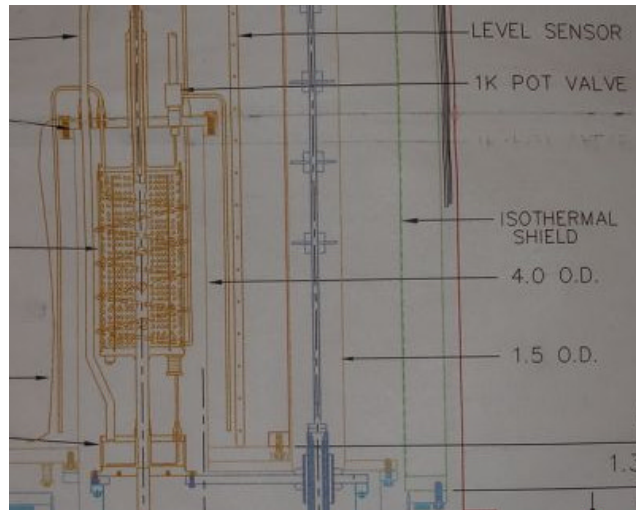
Inside, there is nitrogen reservoir. It is at 77 K and shields the inner reservoir from the room temperature radiation.

The Helium reservoir is at 4.2 K and shields the sample and He-3 systems from the 77-Kelvin-Nitrogen reservoir radiation.

The cryostat also consists of a He-3 insert which includes the charcoal sorption pump, the 1 K pot and the He-3 pot.



The 1 K pot can contain liquid He-4 and is used to condense He-3 gas into the He-3 pot. He-4 is introduced into the 1K pot through the 1 K pot needle valve (1 K pot valve on figure 2). The flow rate of He-4 can be adjusted from the top of the cryostat. When the needle valve is opened, some liquid Helium will be introduced into the 1 K pot. We then pump on the 1 K pot to remove the He-4 vapor, thus causing the liquid



helium to evaporate and re-establish some lower vapor pressure so that the vapor is in equilibrium with the liquid. While evaporating, the 1 K pot will cool down ultimately to approximately 1 Kelvin.

He-3 is condensed into the He-3 pot which is installed below the 1 K pot. The charcoal pump absorbs the He-3 gas from the He-3 gas reservoir in a first step. Then, by heating the charcoal, He-3 gas is desorbed towards the bottom. When the gas is in contact with the 1 K pot which has been cooled down as described above, it will condense and be collected in the He-3 pot. So there will be liquid He-3 at around 3 K. To reach a lower temperature, the charcoal pump is cooled down again. When it is cooled, it functions as a vacuum pump on the He-3 pot and will reduce the vapor pressure of He-3 liquid. So liquid He-3 will evaporate to re-establish the liquid-vapor equilibrium and the He-3 pot will be cooled down. A cooper thermal strap links the He-3 pot and the sample holder. Thus the sample holder reaches also a low temperature.

There are 4 sensors in the cryostat to measure the temperature at specific locations.

TA is a diode sensor that measures the charcoal temperature.

TB is a diode sensor reads the 1 K pot temperature.

TC is a Ruthenium Oxide resistor which gives the temperature of He-3 pot in Kohm.

TD is a Ruthenium Oxide resistor that measures the temperature of the sample holder in Kohm.

For TC and TD, a table is given to convert the measured resistance to Kelvin.

2. The tests

2.1. *The first cool down*

During this experiment, the goal was to determine the lowest temperature that the cryostat could reach. Optical accesses were closed so that the sample holder wasn't affect by a direct heat load from the room temperature. This would tell us if the cryostat works well and hasn't been damaged during its transportation. Since the cryostat was insulated from room temperature and 77K radiation, we were expecting the cryostat to reach a very low base temperature.

After assembling the cryostat (the process is described in the annex), we started the cooling. There were several things to find out during this first cool down, besides the lowest temperature that could be reached: time needed for the pre-cooling and the cooling itself, the hold time, and the rate of evaporation of liquid helium-4.

We pumped the cryostat for several days to make sure that we had a good insulation vacuum. Eventually we had $0,7 \cdot 10^{-3}$. However, this value should be considered with precaution since the vacuum gauge itself may be limited and may not be able to measure below this pressure. We also pumped out the 1 K pot.

Before starting the cooling, we checked whether the cryostat was leak tight thanks to a leak detector. It is combined with a pump and can detect helium gas. When Helium gas is detected, the detector emits a sound. The more it detects Helium gas, the louder is the sound. It was connected to the evacuation valve and was pumping the cryostat. It was obviously emitting a weak sound since there is still some air inside. When we stopped pumping, the sound was almost muted, which meant that helium wasn't going to the leak detector anymore. This proved that the detector was working properly. We flowed Helium gas into the 1K pot and the charcoal circuit in the cryostat and no louder sound was heard: therefore everything was well sealed.

Then we kept flowing helium gas through the 1 K pot and the charcoal circuit to get rid of any residual air which was still in the cryostat. This flow exhausted through the He fill port.

First, we filled the inner and outer reservoir with liquid Nitrogen and let it cool down for some days. When the Nitrogen was running out, the cryostat was filled again. We wanted to make sure that the cryostat was pre-cooled enough before going to the next step.

time (Min)	TA (K)	Notes
0	239,657	1st day; helium reservoir filled with N
5	239,123	
140	120,178	
1160	90,205	19 hours later
1540	85,39	1 days later; N reservoir filled with N
2580	79,818	after an overnight cooling down
2625	79,657	He reservoir refilled
2635	79,606	
2735	79,318	
6855	78,328	almost 5 days later

table 1

In the table 1, we can see that the outer jacket was filled with Nitrogen one day after the beginning of the pre-cooling. In fact, we had some troubles because the transfer line's connectors didn't match with the cryostat. So when we filled it with the liquid Nitrogen, a lot of liquid was wasted. We didn't have enough for the outer jacket, that's why we had to wait for a day to get a new Nitrogen tank. However, this problem has been solved by finding the correct connectors and this shouldn't be an issue for the next cool downs.

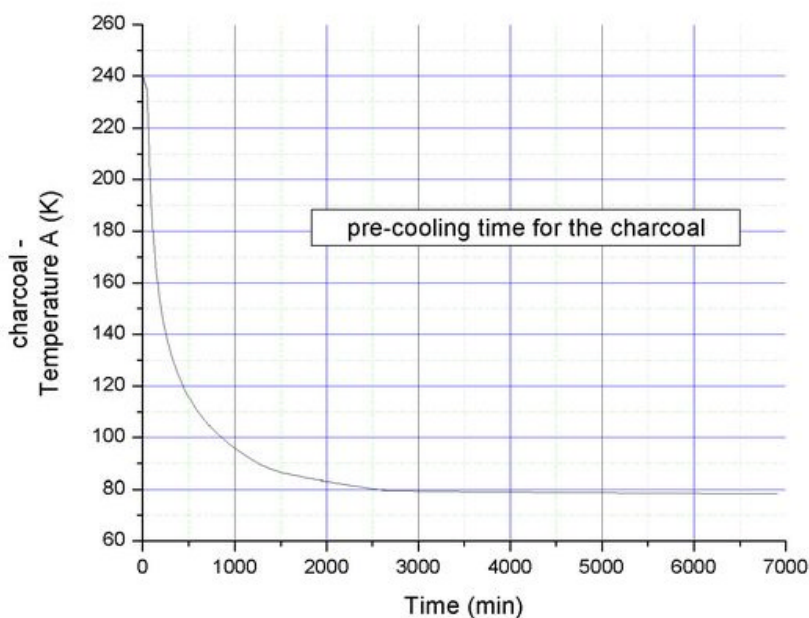


figure 1

Figure 1 shows the evolution of the temperature of the charcoal for 5 days during the pre-cool. But there's no need to wait for too many days. Once the cryostat reaches 80 K, it can be considered ready. Waiting for two days should be enough. In fact, after 2 days (3000 min), the charcoal reached 80 K and then, the temperature is nearly the same after that. It takes more or

less time according to the way the cryostat is pre-cooled. If we didn't pump on the charcoal circuit, it could have taken longer. In this case, we pumped on the charcoal circuit. Nitrogen liquid was flowing through the capillary, and was evaporating inside when it was in contact with the charcoal. So the charcoal was cooled after that. We also tried slowly flowing He gas through the charcoal circuit which provides a thermal link to cool the charcoal.

During the pre-cooling, we also open the needle valve and pumped on the 1 K pot to cool it down using the liquid nitrogen.

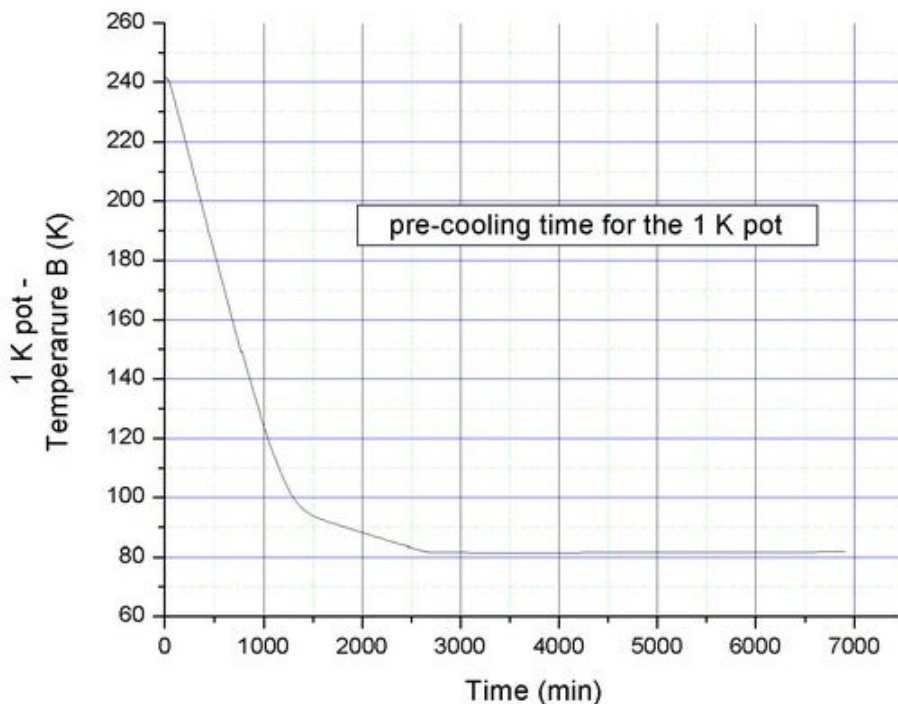


figure 2

Figure 2 shows the evolution of the temperature of the 1 K pot. After 40 hours (2500 min), the 1 K pot reached almost 80 K. It can be cooled down faster if the needle valve is opened wider. Thus more nitrogen is flowing inside the 1 K pot. Obviously, this will use more Nitrogen. The inner reservoir was almost full of Nitrogen. But the level of Nitrogen in this reservoir doesn't affect the time of pre-cooling. Plus, it has to be removed after the pre-cooling, so there's no need to fill it entirely.

The 1K pot and He-3 post need to be pre-cooled overnight. During this process, He-4 gas is flowed into the cryostat via the 1K pot to make a slight overpressure. This gas will behave as an exchange gas and cool down the 1K pot. Indeed, He-4 gas also allows heat exchange and cools the He-3 pot. Otherwise, the He-3 pot is well insulated by vacuum and is not pre-cooled.

time(Min)	TC (K)	notes
0		1st day; helium reservoir filled with N
140	250	
1160	250	19 hours later; overnight cool down
1540	250	1 days later; N reservoir filled with N
2580	95.5	after an overnight cool down, overpressure built
2625	95.5	He reservoir refilled
2735	90	
6855	85	almost 5 days later
6915	85	

table 2

Table 2 shows the evolution of the temperature of the He-3 pot. After the first overnight pre-cooling, the temperature didn't lower. Even though the 1 K pot and the charcoal were cold after 500 min, they didn't cool down the He-3 pot after an overnight pre-cooling. During the second overnight pre-cooling, we flowed some Helium gas into the cryostat so that a small overpressure was made and the He-3 pot started to cool.

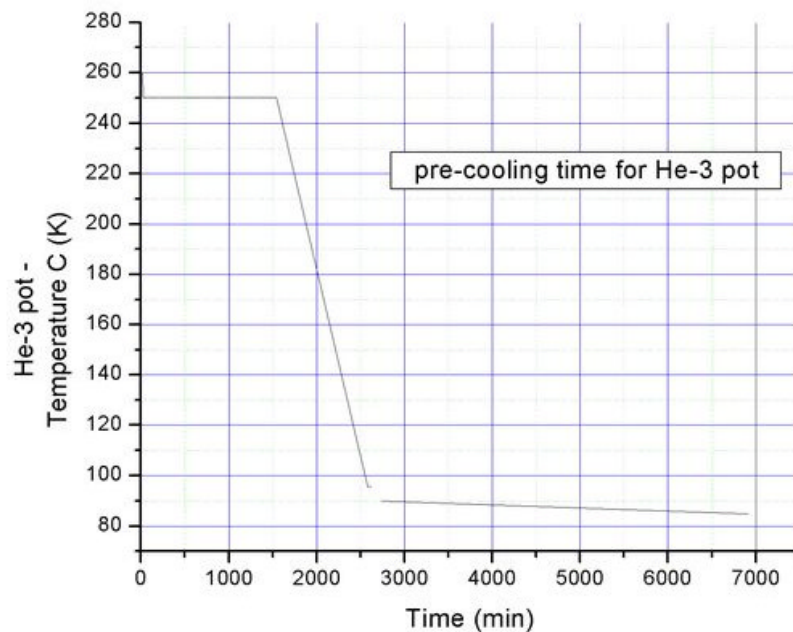


figure 3

Figure 3 show us that the He-3 pot started cooling down after 30 hours (1800 min). So it cooled down during the second overnight pre-cooling. This confirms us that the insulation vacuum is good. The He-3 pot is isolated from the 1 K pot and the inner reservoir and only He-4 gas can cool down the He-3 pot by heat exchange.

When both reservoirs and He-3 pot are cooled, the liquid Nitrogen in the inner one needs to be blown out. When filling the Helium reservoir, maybe it's better to start slowly. The liquid

is going to evaporate, so there's no need to use a lot of liquid helium in the beginning. It is expensive, so we'd better keep it to fill the cryostat when it is running out of helium during condensation or later. That's what we figured out after the 2nd cooling.

When the inner reservoir is full of liquid He, the He-3 procedure can be started. As described earlier, we pump on the 1 K pot while the needle valve allows a small amount of liquid He-4 into this pot. After a while, its temperature is below 2 K. Then, it has to be kept under 2 K. So the needle valve needs to be adjusted to maintain the balance of low pressure and continuous He-4. We tried to close it as much as possible in order to reduce the usage of He-4, but also tried to keep it open as much as required to keep the 1 K pot below 2 K. I didn't do much during this part, Philippe helped me since he has more experience. Indeed, the risk was to assume that the needle valve was enough open to keep the 1 K pot below 2 K, let the process run and notice the day after that it was actually too closed and so the pot had run out of He-4. Then we used the charcoal heater to warm the charcoal to 40 K if it is below this temperature. Thus it will release the He-3 gas toward the colder bottom of the cryostat. This gas will condense when it is in contact with the 1 K pot which is kept below 2 K. The liquid is then collected in the He-3 pot. We let this condensation run overnight. We know that the He-4 gas starts condensing when the He-3 pot cools down until 3 K. This means we have liquid He-3 in it. Eventually, 17 hours passed with the charcoal heater turned on and with the pumping on the 1 K pot. It's not necessary to wait that long. 5 hours is generally enough.

During the condensation of He-3 we estimated the usage of He-4. During 20 minutes, the level of He-4 decreased by 0,18". So we estimated that the He-4 usage was 0,54"/hour. But that 20-minute-measurement wasn't representative for a condensation cycle. However we let the condensation run overnight, so we couldn't note the evolution of He-4. So with the measurements which were taken before and after the night, we estimated that 10,63 inches of He-4 were used during 17 hours. So we had a 0,61"/hour usage.

When all the He-3 is condensed, the heater is turned off and we pumped a little bit on the charcoal circuit to cool it down. The charcoal then pumps on the He-3 liquid in the He-3 pot which then reaches a low temperature.

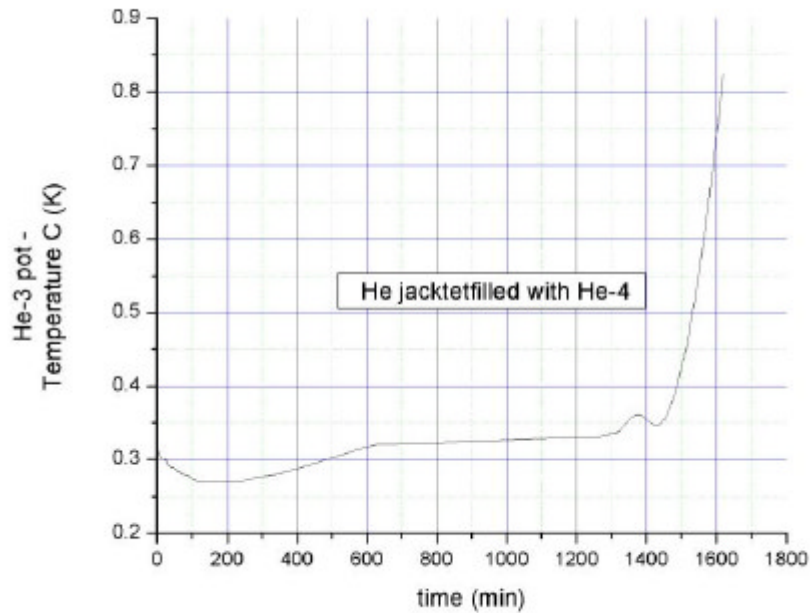


figure 4

Figure 4 shows the holding time once liquid He-3 reached a low temperature. We reached around 0,3 K or below some times for nearly 24 hours. Moreover the hold time is mainly due to the running out of liquid He-4, probably because of the previous 17-hour-wait. If it was still enough, we could have held it longer. It is risky to refill the He-4 rapidly since the transfer can blow out the remaining liquid He-4 in the cryostat. Also, when the He passes through the transfer line, it will warm and then further evaporate the He-4 in the cryostat. What I was advised to do is to lift the He-4 filling rod, blow gently He-4 so that it will cool the transfer line first, and then refill the cryostat.

time (min)	TC	height	Notes
0	0,31	6,5	1st day
5	0,31	6,47	--
25	0,3	6,37	--
65	0,28		--
90	0,28	6,02	--
110	0,27	5,92	--
245	0,27		--
285	0,278	6,28	--
1265	0,33	0,27	--
1295	0,335	0,02	--
1430	0,33		2nd day
1490	0,385		
1620	0,825		

table 3

Regarding the He-4 usage while the cryostat reached its lowest temperature, we had in average a 0,25"/hour usage. During this cooling, we let the cryostat cool overnight during the final step. So we don't know how long it takes to reach 3 K when the He-3 gas started condensing.

The first cooling was successful. We reached 0.3 K and we could hold it for nearly

24 hours. However, we were running out of He-4 and this probably limited the holding time to less than 24 hours. This showed us that the cryostat worked and wasn't damaged during its transportation. However, this current configuration is useless for optical experiments. Optical accesses are closed, so no spectroscopy can be carried out. What is interesting now is to cool down the cryostat in a real situation, that is to say, using windows and see what the new results are.

2.2. The 2nd cool down

In this new experiment, we used polyethylene windows (figure 5). Their sizes were quite large, and we used them on both side of the cryostat as if we were carrying out a transmission measurement. This is the worst case for heat load, and we wanted to know the cryostat's limit. Polyethylene windows transmit light below 700 cm^{-1} (wavenumbers). During the cooling, we did 3 attempts to He-3 temperatures. However, I met some difficulties since I handle the cooling down mostly by myself. The first time, the cooling failed. The second and third times were successful. During the 2nd attempt, aluminium was folded around the outer window. We thought this might decrease the effects of room temperature radiation. In the third try, we removed the aluminum.



polyethylene windows

2.2.1. first attempt

The overall process was the same as the previous cooling. I was running out of liquid Helium-4 before the end of the experiment. I can't really explain what happened, but here are some assumptions. The first one is that the He-4 tank wasn't completely full. Or else, when I started filling the inner jacket with He-4 liquid, the He-3 pot wasn't cold enough, so it took longer to condense the He-3 gas and then the evaporation of liquid He-4 was faster than it should have been. I may have also adjusted the needle valve badly leaving it too far open.

time (min)	TC	notes
0	115	inner jacket filled with He; pump on 1K pot; He through charcoal
90	113	close charcoal; open needle valve
105	113,5	--
120	111	charcoal heater ON; refilled with He
135	110	aluminium folded around windows
165	97	--
195	96	--
275	90	--
350	87	close all ports; still pumping 1 K pot
380	86	--
385	80	--
865	2,3	8 hours later
1345	217	8 hours later; experiment failed

table 4

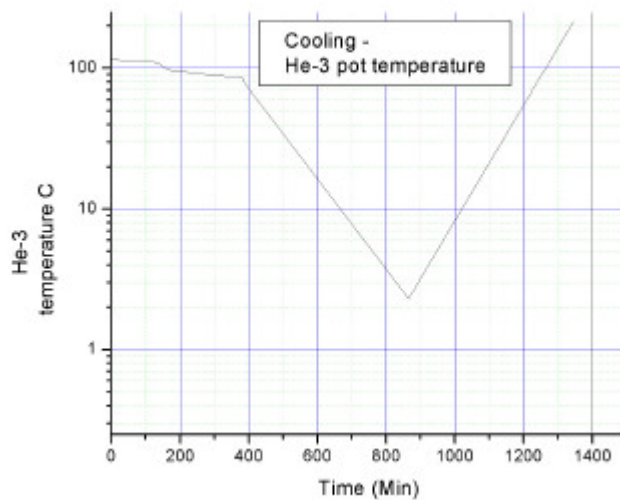


figure 5

Table 4 and figure 5 show the evolution of the He-3 pot temperature. It isn't entirely accurate since we didn't have enough data points. We started to fill the He-4 reservoir when the He-3 pot temperature was at around 130 K. During the first cool down, its temperature was around 80 K when we started transferring He-4. That may explain why we were running out of liquid

He-4 since it uses up more He-4 to cool from 130 K. According to the firm Janis, we need at least 7 hours when the He-3 pot is at 130 K. Last time, when it was at 80 K we waited 17 hours but 5 hours seemed to be enough. Moreover, if we assume that all the He-3 gas condensed after 7 hours, we didn't turn off the charcoal heater since the process begun in the evening. So when we came later to check the cryostat, we had 2.3 K at the He-3 pot, but it couldn't get lower since the charcoal was still at 40K and so wasn't pumping on the liquid He-3.

2.2.2. second attempt

Eventually, the cryostat started to warm up since the He-3 pot and He-4 reservoir was empty. However it was still cooled enough to not require another pre-cool, so we decided to re-cool it a second time as soon as possible. This time, the process was quite successful.

Time	TC	TD	Lhe-4 level (inches)	Notes
2345	86	86	15.46	1k pot < 2 K, charcoal heater ON; adjust needle valve
2405	85		14.4	--
2885	4.5	6	10.39	--
2890	2.8	4.2	10.27	--
2920	1.95	4,1	9.8	--
2930	1.8		9.65	TC different of TD; already observed last time
2945			12.16	refill inner jacket with He-4
2990	1.55		18	Refill
3590	1.65	--		overnight; 10hours later; charcoal heater OFF
3630	1.55	4	10.76	--
3660	1.45		9.4	flow meter
3715	0.99		9.26	--
3730	35			open flow meter more
3740	2.8			Switch back to pumping on the charcoal circuit
3775	0.55			

table 5

However, we noticed some strange behaviour. The He-3 pot and the sample holder are supposed to be thermally well linked. But their temperature was different. This was probably due to the heat load on the sample holder from the room temperature radiation. It was already observed in the first attempt before we were running out of liquid He-4 (look at the tables in annex). The transmission of the 300K radiation through the polyethylene windows prevent it from cooling properly, even with aluminium folded around it. In fact, all the outer structure is at 300 K, and radiates to the colder inner parts of the cryostat. The coldest stages have further radiation shielding at 77K, 4.2K and even 1K, except for the sample holder which through the polyethylene windows has a direct view of the 300K radiation. So, even though the aluminium outside the polyethylene windows, the windows themselves are at room temperature and so radiate towards the sample holder.

Moreover, when the charcoal was cooling down; the He-3 pot was reaching a low temperature and then increased suddenly to 2.8 K. We concluded that the charcoal wasn't pumping

anymore so, the vapor-liquid equilibrium was re-established. We were using the flow meter instead of pumping on the charcoal circuit to cool down the charcoal. But it seems that it is not efficient enough with the heat loads coming through the polyethylene windows.

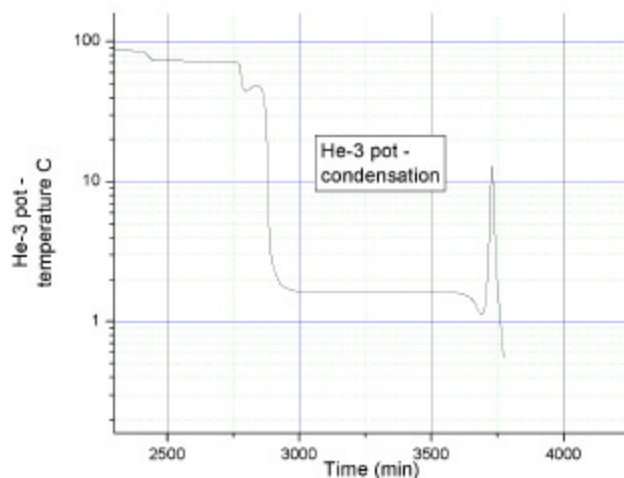


Figure 6

Figure 6 shows the evolution of the He-3 pot during condensation. When we used the flow meter to cool down the charcoal, we were reaching a low temperature and but it started to warm up at around 3700 min. So we removed it and pumped on the charcoal circuit. When the lower temperature was reached, we waited for a while and we decided to remove the aluminium, and no more than three minutes later, the temperature went up suddenly. However, maybe this was a coincidence and the He-3 was already evaporated because the holding time was short in this case.

As a consequence, we wanted to know what the hold time is for this low temperature with the polyethylene windows with and without the aluminium wrapped around the outer windows.

We re-condensed the He-3 by heating the charcoal. Usually, it is required to wait 4 hours after the He-3 pot reaches around 1.8 K but since the cryostat has been cooled before and that we had already condensed the He-3 earlier, it wasn't necessary to wait that long. In this case, I just waited for 2 hours.

Time (min)	TC	TD	height	notes
0	30	30		--
5	2,8	5		--
15	2,8	5		charocal heater ON
30	1,85	4,2		--
120	1,73	4,2	7	--
125	1,66	4	6,75	--
135	1,66	3,8	6,67	--
145	1,66	4	--	--
155	1,66	4	--	--
165	1,66	4	6,48	start pumping on charcoal
180	1,3	3,9	6,35	--
185	1,05	3,8	6,31	--
190	0,76	3,5	6,29	--
195	0,63	3,5	6,25	--
205	0,56	3,5	6,17	--
225	1,9	4	--	--

table 6

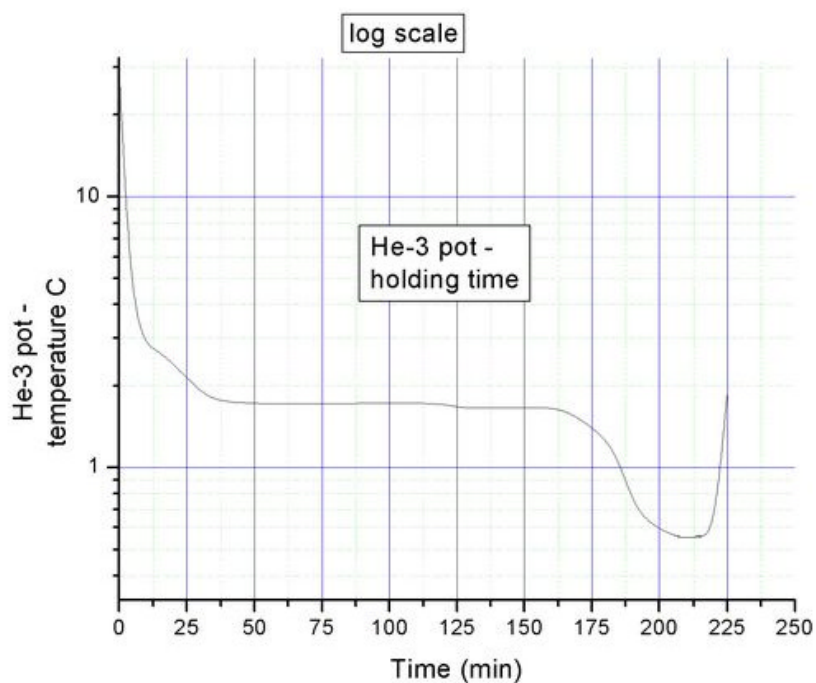


figure 7

Figure 7 shows the holding time with aluminium foil wrapped around the polyethylene windows. Once the lowest temperature seemed to be reached, I measured that the holding time was around 30 minutes. This effectively showed that the He-3 liquid was already evaporated when the Aluminium was removed in the previous condensation run.

2.2.3. third attempt

After this test, we wanted to measure the holding time without the Aluminium around the windows. We re-condensed the He-3. The holding time was nearly the same. The He-3 pot reached nearly the same temperature as previously, which is consistent.

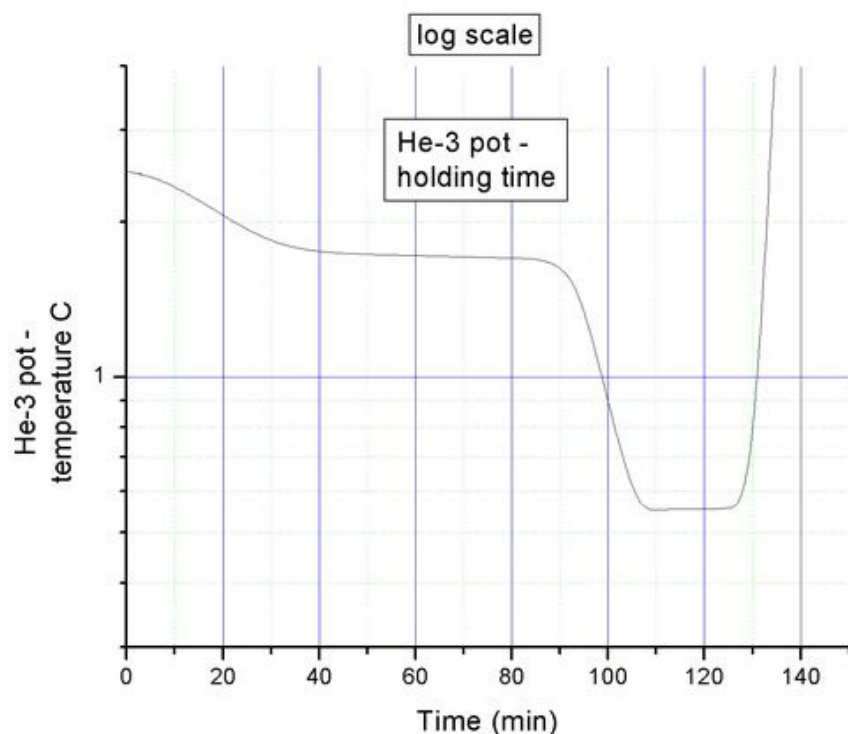


figure 8

The Aluminium does not affect the hold time or base temperature. Once the He-3 is condensed, the He-3 pot and sample holder reached essentially the same temperature with or without the aluminium. This wasn't really surprising since the hold time was nearly the same. However I was expecting a higher temperature, and a shorter hold time without the aluminium. It came out that the outer windows don't block the radiation. All the outer cylinder is at the room temperature. So it emits radiation to the 2nd window. The first window is supposed to cut out transmitted radiation above 700 wavenumbers, but it is also a 300K emission source itself.

Finally, these results are still relevant for our understanding of the cryostat's performance. So now we want to know if the hold time measured is only due to the heat load on the sample holder from this 300 K radiation or if there are further unknown heat loads at play (such as bad thermal connection in the cryostat or a part of the sample holder touching a higher temperature radiation shield). Consequently, I tried to calculate the heat load from the 300K radiation. This will tell us the predicted holding time which can be obtained and we will compare to our measured holding time measurements.

3. Calculation of heat load

Calculating the heat load was probably the hardest part of my work. I can't say if they are completely accurate. However I'll try to give the reasoning behind these calculations. Thus, anyone who wants to work on this cryostat may still improve upon them.

These calculations, which are supposed to confirm the results of the second cooling, are then generalized. Then, we can tell what heat load is expected for each type of window. This can also tell us for how long we can hold the lowest temperature and what is the difference of temperature between the sample holder and the He-3 pot is predicted to be.

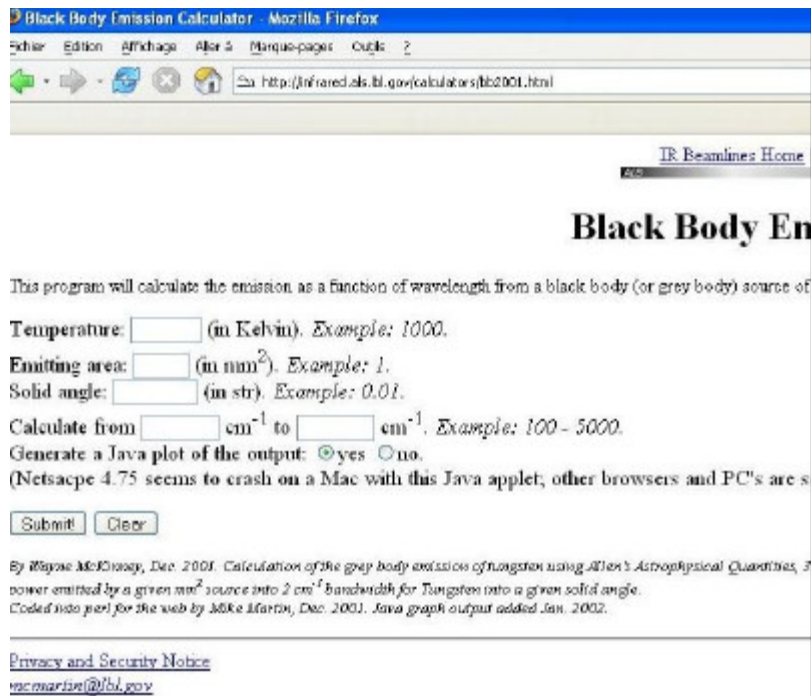
3.1. first calculation

According to my results and data given by Janis, I found that the energy needed to heat the 30 liters of He-3 gas in this cryostat's reservoir from a condensed liquid He-3 at 0,55 K to 3.2 K is around **45 J**.

To calculate the heat load, I used the blackbody theory since in physics, blackbody is a model used to calculate an object's thermal radiation as a function of its temperature. A blackbody absorbs all lights it receives and radiates energy whose spectrum depends on its temperature.

The entire integrated energy density radiated by a blackbody is $\sigma \times T^4$ (where $\sigma = 5,67 \cdot 10^{-8} \text{ W/m}^2/\text{K}^4$) that is to say **459 W/m²** for T=300 K.

However, the sample holder doesn't receive all the radiation since the inner windows will block some wavelengths. We only have to take in account a fraction of the spectrum. To estimate the fraction of power transmitted, I used a program coded by Mike Martin which was available on the beamline1.4.3's website: <http://infrared.als.lbl.gov/calculators/bb2001.html>



The screenshot shows a web browser window titled "Black Body Emission Calculator - Mozilla Firefox". The address bar shows the URL "http://infrared.als.lbl.gov/calculators/bb2001.html". The page content includes a title "Black Body En" and a description: "This program will calculate the emission as a function of wavelength from a black body (or grey body) source of". Below this are input fields for "Temperature:" (in Kelvin, Example: 1000), "Emitting area:" (in mm², Example: 1), "Solid angle:" (in str, Example: 0.01), and "Calculate from:" (in cm⁻¹, Example: 100 - 5000). There is a checkbox for "Generate a Java plot of the output:" with "yes" selected. Below the inputs are "Submit" and "Clear" buttons. At the bottom, there is a copyright notice: "By Wayne McKinney, Dec. 2001. Calculation of the grey body emission of tungsten using Allen's Astrophysical Quantities, 3rd edition, 1973. Coded into perl for the web by Mike Martin, Dec. 2001. Java graph output added Jan. 2002." and a "Privacy and Security Notice" link.

For a blackbody at 300 K, the power of the radiation for photon energies above 4000 cm^{-1} is negligible compared to the emission for energies below 4000 cm^{-1} . With the program, I estimated the emission from a blackbody at 300 K for energies between 1 and 4000 cm^{-1} .

We know that the polyethylene window transmits light below 700 cm^{-1} . So I also calculated the emission from a 300K blackbody for energies from 1 cm^{-1} to 700 cm^{-1} .

In both cases, I used the same values for all other input parameters. As a result, I can determine the fraction transmitted, and we can estimate the energy density which goes to the sample.

So, for a blackbody source of size 1 mm^2 and temperature 300 K which emits into a solid angle of π str from 1 to 4000 cm^{-1} , it emits $4,59 \cdot 10^{-4} \text{ W}$.

Then, for a blackbody source of size 1 mm^2 and temperature 300 K which emits into a solid angle of π from 1 to 700 cm^{-1} , it emits $2,16 \cdot 10^{-4} \text{ W}$, that is to say **47% of $4,59 \cdot 10^{-4} \text{ W/m}^2$** .

So in our case I have to take in account only 47% of $S \times T^4$ from 300K makes it to the sample.

However, the sample holder won't receive all this fraction of energy since the sample and the emitting area at 300 K are separated by a certain distance and the sample sees only a certain solid angle from the emitter. That's why a view factor has to be introduced to take in account the fraction of power leaving the surface at 300 K (object 1, radius r_1) and reaching the sample holder (object 2, radius r_2). Usually, one can find these geometrical view factors tabulated in a book, but it wasn't available in the laboratory. Some formulas can estimated the view factors, especially when the two objects have a circular geometry. In this case, if we assume that the window and the sample holder have a circular form, we are close to reality.

To estimate the view factor F_{1-2} , I used the following equation:

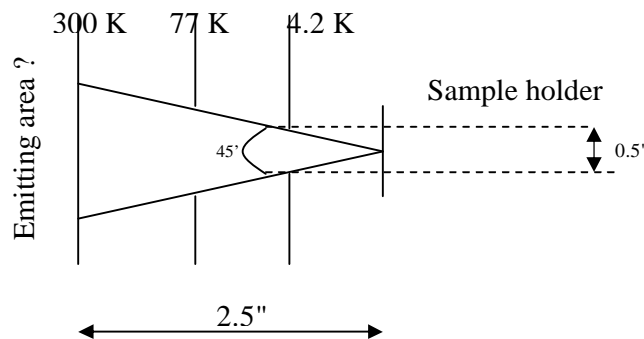
$$R = \frac{r}{d}$$

$$X = 1 + \frac{1 + R_2^2}{R_1^2}$$

$$F_{1-2} = \frac{1}{2} \left(X - \sqrt{X^2 - 4 \left(\frac{R_2}{R_1} \right)^2} \right)$$

where d is the distance between the emitter and receiver, r_1 is the radius of the emitter and r_2 is the radius of the receiver (details of form factor formula in the annex). The area of the receiver was estimated as a disc with a radius of **0.25 inches**.

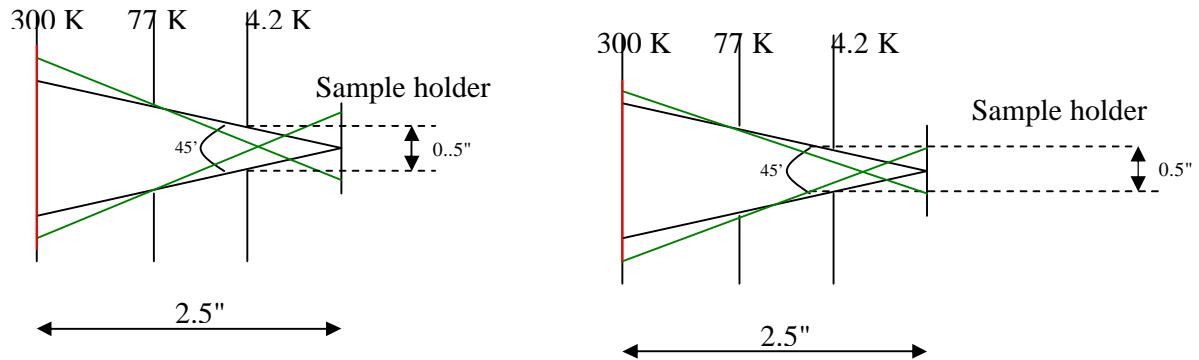
Then we estimate the area of the surface which radiates at 300K.



We find that the emitting area is a disc with a radius of **1 inch**.

But this area only radiates to the centre of the sample holder.

All the area of the sample holder receives radiation, so the emitting area should be a bit larger as is shown below by the green rays and the resulting red emitting area:



We estimated it as a disc with a radius of **1.1 inches**.

We notice that the area of the receiver may be bigger than the inner window diameter as shown by the green rays above, but we won't take this into account.

So:

$$\begin{aligned} r_1 &= 1.1'' \\ r_2 &= 0.25'' \\ d &= 2.5'' \end{aligned}$$

Finally, we have **$F_{1-2}=0,024$**

Then I can calculate the power received by the sample holder following the formula below:

$$P = F_{12} \times R \times \epsilon \times T^4 \times A \times 0,9$$

and 0,9 is the coefficient of emissivity of the window since it's not a real blackbody.

R is the fraction of power which is transmitted through the window, here 47%.

A is the area of the emitter.

So, **$P=7,6 \text{ mW}$**

The energy received by the sample holder after a time t can be estimated:

$$P \times t = E$$

This energy received by the sample holder will be transferred to the He-3 pot. After a time t, it should be equal to **45 J**.

For the polyethylene window, the calculation shows we can hold the low temperature during one hour and a half. However during the experiment I had a **30-minute-holding time**. There are probably other heat loads that I didn't take in account like the 77 K radiation from the window at 77 K. Some approximations were made about the windows, so the view factor may be different than what it was estimated.

However, I could compare those calculations with the hold time it was obtained in Boston when Jason Singley and Zhao Hao went there and see whether there are still consistent. They used 2 sets of quartz windows. So they had the same configuration as mine. However the sizes of the windows were different. The outer window and the central window are 0.25-inch-radius. The inner window is bigger; the area seems to be the double of the outer window.

The dimensions I chose for the emitter and the receiver are those below according to the same reasoning:

radius of emitter: **0.5"**
radius of receiver: **0.4"**

I found a hold time of 166 hours. When they ran the experiment in Boston, they could hold around 100 – 120 hours. The difference is smaller than for my experiment, but the theoretical result is still superior at the experimental value. So others heats loads are missing.

Even if my calculations are still approximate, it is possible to figure out the potential hold time for different windows. Obviously, those results should be interpreted with caution since not all heat loads are taken in account. However, they can give an idea of what we can have. In annex, we can find the C program I coded to obtain those followings results. When the program is executed, we just need to enter the dimension of the emitter and receiver and the fraction of heat which is transmitted.

We had two sizes of windows retainers: the big ones I used for my experiment and the windows retainers use in Boston which are smaller.

The beamline 1.4.3 owns different windows which cut-off the light above different energies: 700cm^{-1} , 200cm^{-1} , 100cm^{-1} , 50cm^{-1} , 20cm^{-1} and below 100cm^{-1} and over 2500cm^{-1} for the quartz. With the program in the website, the ratio of power radiated through each window is respectively: 47%; 3%; 0,4%; $7.10^{-4}\%$; $5.10^{-5}\%$; and 0,007%.

He-3 hold Time

	700 cm^{-1}	quartz	200 cm^{-1}	100 cm^{-1}	50 cm^{-1}	$20\text{ cm}^{-1<}$
big windows	1h30 exp: 30min	109h	25h	191h	Not realistic	not realistic
small windows	2h30	166h exp: 100h	43h	320h	Not realistic	not realistic

Temperature differential between sample and He-3 pot

	700 cm^{-1}	quartz	200 cm^{-1}	100 cm^{-1}	50 cm^{-1}	20 cm^{-1}
big windows	1 K exp:2,95K	0,01 K	0,06 K	0,008 K	not realistic	not realistic
small windows	0,6 K	0,009 K exp ~ 0,01	0,04 K	0,005 K	not realistic	not realistic

Those results from the calculation are really optimistic. Not all heat loads are taken in account and some assumptions were made regarding the dimensions of the windows. They give an idea of the potential hold time and the real hold should be divided by 2 or 3.

This part may be useful if we use windows retainers other than those 2 which were given.

However, we may be able to calculate a more accurate hold time once we know the hold time for a type a window with a fixed size.

3.2. second calculation

In the case of the big windows, we did an experiment with the polyethylene windows. So we know the holding time, and then we can estimate the power of all heat sources received by the sample holder.

P=0.025W

Assuming this is all radiation heat load, according the formula

$$P = F_{12} \times R \times \sigma \times T^4 \times A \times 0,9$$

We can estimate the term $F_{12} \times A \times 0,9$.

Even if we change the type of windows, this term won't change if we keep the same windows retainers.

So we can replace $F_{12} \times A \times 0,9$ by the experimentally determined value to give us a more realistic holding time.

We coded another C program (in the annex) which give us the results following this reasoning.

He-3 hold time

	700 cm ⁻¹	quartz	200 cm ⁻¹	100 cm ⁻¹	50 cm ⁻¹	20 cm ^{-1<S}
Big windows	30 min	33h	7h	58h	Not realistic	not realistic
Small windows	1h30	100h	23h	175h	Not realistic	not realistic

Temperature differential between He-3 pot and sample holder

	700 cm ⁻¹	quartz	200 cm ⁻¹	100 cm ⁻¹	50 cm ⁻¹	20 cm ^{-1<S}
big windows	3,1 K exp:2,95K	0,04 K	0,2 K	0,02 K	Not realistic	not realistic
Small windows	1,04 K	0,02 K exp ~ 0,01	0,07 K	0,009 K	Not realistic	not realistic

Sample base temperature

	700 cm ⁻¹	quartz	200 cm ⁻¹	100 cm ⁻¹	50 cm ⁻¹	20 cm ^{-1<S}
big windows	3,4 K exp:3,5K	0,31 K	0,47 K	0,29 K	0,27 K	0,27 K
Small windows	1,31 K	0,29 K exp ~0.27 K	0,34 K	0,28 K	0,27 K	0,27 K

These results seem to me more realistic. However different sample mounts may have other heat loads. The non-realistic results for windows which transmits light only under 50 cm^{-1} and 20 cm^{-1} , are because the 300K radiation gives a negligible heat load and other residual heat loads in the system will dominate the experimental hold times. Indeed, windows which cut at 50 cm^{-1} and 20 cm^{-1} transmit low power radiation from 300 K.

Conclusion

My project during this internship was to test the He-3 cryostat and understand its method of cooling so that I could write down a manual for future users. I started with a basic test; the cryostat was well isolated from room temperature. The cryostat could reach a very low temperature: $<0,3\text{ K}$ and we could hold it for at least a day. It could be longer if we were not running out of He-4. But this configuration is useless in practice since no spectroscopy can be done. Then I tested the cryostat with large polyethylene windows which transmits light below 700 cm^{-1} wavenumbers. The resulting performance of the cryostat was not that good since this is one of the worst cases possible for radiative heat loads. Then I figured out a calculation from this experiment to estimate the head load through the window and which confirm our results. I generalized this calculation so that we can estimate the hold time and the lowest temperature which can be reached with several windows which transmits at different wavelengths.

Concerning the project I had, it was interesting to discover something else that I was taught in my engineering school such as electronics or spectroscopy in x-ray region. The work in itself was also interesting in different point of views. Handling the cryostat allows me to learn some techniques in the cryogenic field. Also, another interesting part was that I started from scratch. Indeed, the cryostat was still sealed in its box when I arrived and I had to think about several things like designing some pieces to hold the cryostat safely when it was run, designing a manifold so that it will be easier to cool it down for the next coolings.

Moreover, working on a concrete problem made me learn different things. Even though that was something new for me, I could use tools I knew to find solutions like the C program I coded to figure out the different hold times for each window. I could understand the principle of a cryostat thanks to what I learnt 2 or 3 years ago in thermodynamic. I've already known it before but here, I could see for real that besides the tools we learn in our studies, what is really useful is the scientific approach we acquired to solve problems. As for the calculations of heat load, I started by the analogy of the blackbody source at 300 K with a radioactive source and tried to estimate the angle solid in which it radiates the power of radiation. Then, talking with people and then exchanging ideas, I came up to the view factor.

This internship was a great experience. It was a real pleasure to work in this ALS at the beamline 1.4. I could work with nice people in a good atmosphere. Carrying out this internship in a foreign country allows me to be immersed in a different culture and faced the problem of living and working entirely in a non-native language, English. I could meet several interesting people in and out of the lab such as researchers, students in physics, but also other people not connected with science.

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Websites:

http://www.efunda.com/formulae/heat_transfer/radiation/view_factors.cfm

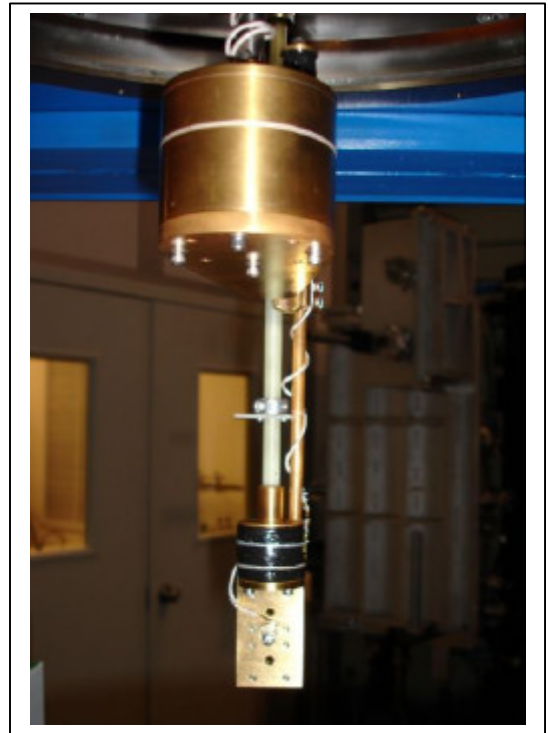
<http://hyperphysics.phy-astr.gsu.edu/hbase/hframe.html>

www.janis.com

ANNEX

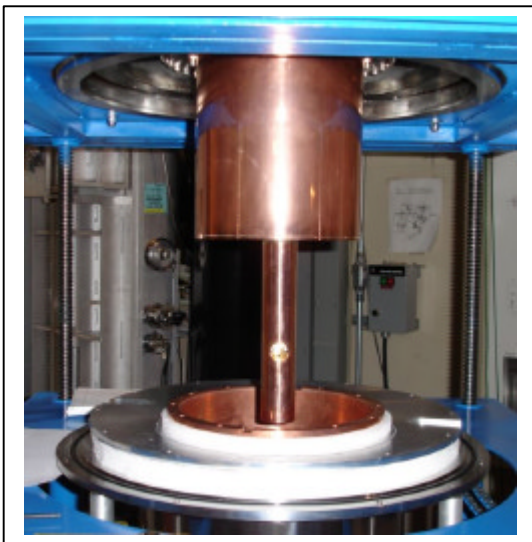


Assembling the cryostat

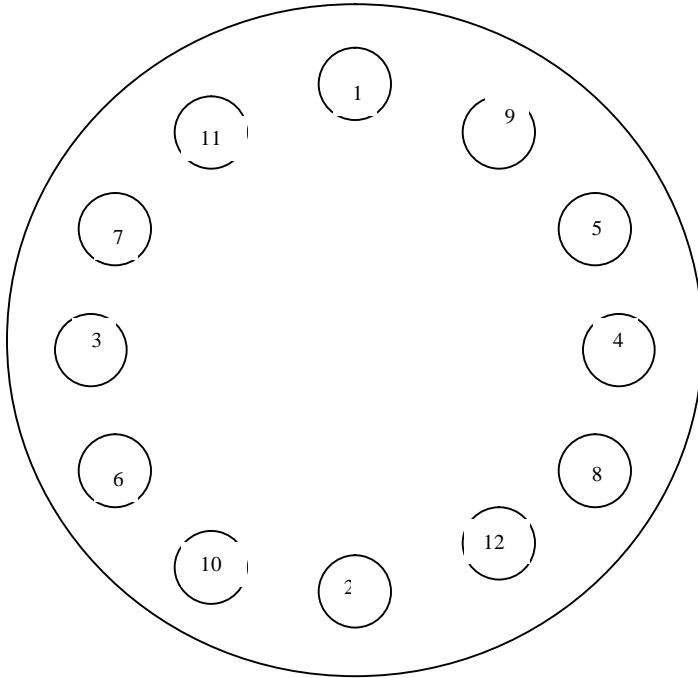


To assemble the cryostat, it is advised to wear gloves so that the inside of the cryostat remains clean.

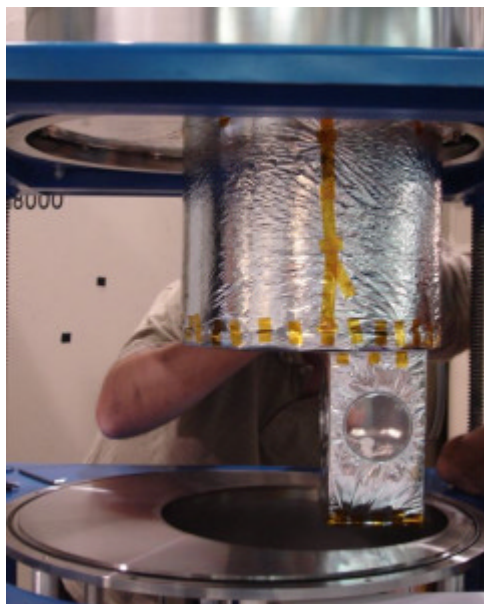
Slowly assemble the both parts since the sample holder just fits in the tail.



When the first cylinder is mounted, look out to let the wire in the hole. During the bolting, the ideal way to do it is to bolt each bolt in 3 shots, choosing the opposite one each time.



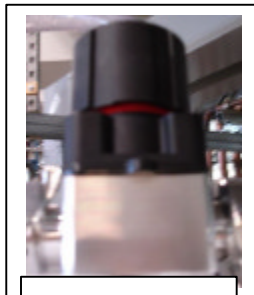
Assemble the second cylinder and the third one in the same way.



Cooling the cryostat



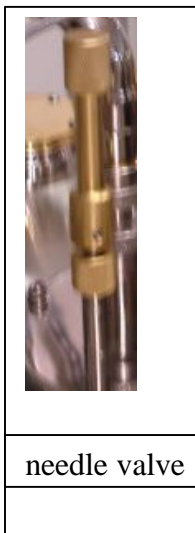
charcoal valve



1 K pot valve



He fill port



needle valve



He transfer line which goes to the He fill port

Insulation vacuum: $0,7 \cdot 10^{-4}$ Torr to $1,1 \cdot 10^{-3}$ Torr

Anytime you open one of the helium fill ports, flow some Helium gas through the 1 K pot and the charcoal circuit to avoid accumulating water and freeze out in the capillaries.

1. Flow Helium gas through the charcoal cooling control valve and the 1 K pot pumpout to clean the inside of the cryostat. The gas should go out by the safety pressure relief valve or the Helium fill port if this one is opened.
2. Flow Helium gas through the charcoal valve and through the 1 K pot pumpout port with the needle valve open in order to prevent both capillaries from accumulating water vapor and eventually freeze out during the cooling.
3. Fill the He reservoir with liquid Nitrogen to start cooling the cryostat. You don't need to fill the whole He reservoir. One half of the He4 reservoir volume should be enough. Close all the ports.

4. Fill the whole Nitrogen reservoir with liquid Nitrogen to cool down the cryostat.
5. To accelerate the cooling, you can first open slightly the needle valve (V1) and pump on the 1 K pot. Pump also on the charcoal circuit. Stop the process before reaching 100 K for both of the 1K pot and the charcoal temperatures. You don't want to accumulate liquid Nitrogen in the 1 K pot. ***You want the 1 K pot and the He-3 pot to reach respectively around 100 K and 130 K. It's better if the He-3 pot temperature is below 130 K. Try to let it reach the lowest possible temperature: 85 K. As a result, when you start condensing the He-3, the He-3 pot will collect liquid He-3 faster***
6. Stop pumping on the needle valve and the charcoal circuit if you didn't do so yet. Establish a Helium gas flow through the charcoal pump until overpressure is measured at the safety pressure relief valve. **All other ports closed.** This will make sure that the charcoal capillary is not frozen. Repeat the procedure for the 1 K pot to make sure the 1 K pot capillary is not frozen. Close the needle valve. Once these checks are done, close the He4 gas line (at the cylinder). Make sure there is approximately 1 Bar of He4 in the 1K pot. This exchange gas increases thermal coupling between the 1 K pot and the He3 stage.
7. Check if there is some liquid nitrogen in both reservoirs: firstly, be sure everything is closed. Then open the valve which will allow the pumping on the charcoal circuit. Then, if the temperature of the charcoal is decreasing, this means you have some liquid nitrogen. Close this valve and open the one which will allow the pumping on the 1 K pot. The temperature of the 1 K pot should also decrease. Close the valve.
8. Re-establish a Helium gas flow through the charcoal pump until overpressure is measured at the safety pressure relief valve. Allow the cryostat to cool overnight.
9. If not already in place, mount the He4 transfer line. Push the liquid Nitrogen out of the Helium reservoir by applying overpressure of Helium gas through the charcoal cooling control valve (V1) and the 1 K pot pumpout (V5).
10. Begin transfer of liquid He into the helium reservoir, as soon as the He4 level in the cryostat is larger than 3 inches, stop the He4 gas flow through the 1K pot and the charcoal lines. Open the needle valve and pump on the 1 K pot.
11. When the 1 K pot temperature is below 2 K, set the temperature of the charcoal sorption pump at 40K with the temperature controller. Adjust the needle valve: try to keep it as close as possible to limit the usage of He-4 liquid, but try to keep it as open as possible too so that the 1 K pot is always below 1 K pot. You should come and check it several times before being sure that the adjustment is correct.
12. When the He-3 pot reaches 1.8 K, wait for approximately 4 hours to be sure that all the He-3 gas is condensed.

13. Turn off the charcoal heater and pump slightly on the charcoal circuit. This will make the charcoal to pump and force the evaporation of the He-3 liquid. The base temperature will reach low base temperature within an hour.

Observations concerning the 2nd cooling

We observed some condensations on the outer windows. However, when we separated the cryostat and checked the inside, we thought that the screws of the 300-Kelvin-window could touch the 77-kelvin-reservoir. So we changed the screws. Screws of the others windows have been changed before and don't touch the reservoirs.

When we were heating the charcoal to condense He-3, we noticed that flowing Helium gas into the cryostat makes the heater to use less power.

In fact, when we stopped flowing Helium, the heater needed more power to warm the charcoal.